

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

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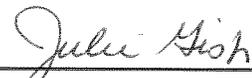
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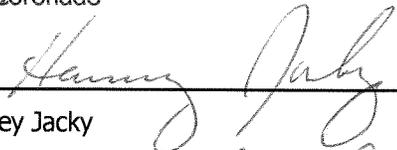
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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

It is a policy at CAS that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. The Quality System is established to meet the requirements of The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP).

CAS maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

CAS is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, CAS also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2*, USEPA, 2001; and *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5*, USEPA, 2001.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of CAS:

"The mission of Columbia Analytical Services, Inc., is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, CAS minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories (2)

- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
 - Separate sample preparation laboratory
 - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. On-time performance, report quality, training, and Quality Assurance are a few of the items that are used to assess performance from an external perspective. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

CAS utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.4 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL)*. The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled CAS documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

CAS maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH)*.

4.5 Subcontracting

Analytical services are subcontracted when CAS/Kelso needs to balance workload or when the requested analyses are not performed by CAS/Kelso. Subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories Outside of CAS Network (ADM-SUBLAB)*. The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.6 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The procedures for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR)*. Also, refer to section 10.4 for a discussion of reference materials.

5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.
- It is the responsibility of all CAS employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the CAS *Confidentiality and Conflicts of Interest Employee Agreement* and the CAS *Commitment to Excellence in Data Quality Policy*. All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

CAS makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the CAS Employee Handbook. This includes the CAS Ombudsman Program, the CAS Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the CAS laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 130 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the CAS/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.
- The responsibility of the **Quality Assurance Manager (QAM)** is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the CAS Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

The Chief Quality Officer (CQO) is responsible for the overall QA program at all the CAS laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each CAS laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).
- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to CAS' EH&S Director.
- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.
- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS provides are properly executed and satisfy the requirements of the client.
- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.
- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.
- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

**Table 6-1
 Summary of Technical Experience and Qualifications**

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	30	Laboratory Director
Julie Gish, M.S.	18	Quality Assurance Manager
Lynda Huckestein, B.S.	20	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	19	Metals Department Manager
Harvey Jacky, B.S.	20	General Chemistry Department Manager
Gregory Salate, Ph.D.	9	Extractions Department Manager
Jeff Grindstaff, B.S.	20	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	18	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	27	Environmental Health and Safety Officer
Mike Sullivan, B.S.	8	CAS Information Technology Director
Lee Wolf, B.S.	23	CAS Chief Quality Officer
Steve Vincent, B.S.	33	CAS President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. CAS management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

CAS has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the CAS Software Quality Assurance Plan (SQAP). The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of configuration management plan are policies for controlling the software version that is in use in the laboratory.

7.2 IT Support

The local CAS Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, CAS corporate IT provides support for network-wide systems. CAS also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

CAS has various systems in place to address specific data management needs. The CAS Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system is used to establish and define sample identification, analysis specifications, and provide a means of sample tracking. This system is used during sample login to generate the internal Service Request. The Service

Request provides a summary of client information, sample information, required analyses, work instructions, deliverable requirements and other necessary information provided on the chain of custody. The LIMS also is the basis for valuable sample tracking mechanisms used throughout the laboratory. Laboratory analysts generate responsibility reports from the LIMS and perform internal chain of custody via the LIMS.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

CAS laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non-CAS access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. CAS uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. CAS recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

CAS uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

CAS routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at CAS and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to CAS. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When CAS ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures for maintaining the chain of custody of the sample(s) (*SOP for Chain of Custody for Sample Transfer between Laboratories [SOP ADM-COC]*), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Once samples are delivered to the CAS sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. CAS has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. CAS also has seven sub-zero freezers capable of storing samples at -20°C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

CAS adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual*. All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at CAS.

Facility security and access is important in maintaining the integrity of samples received at CAS/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The CAS facility is equipped with an alarm system and CAS employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC)*.

8.4 Project Setup

The analytical method(s) to be used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the LIMS generated Service Request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION^a	MATRIX^b	CONTAINER^c	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ^d	6-24 hours ^e
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days ^{EPA}
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days ^{EPA}
Ammonia (SM 4500NH ₃)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO ₃ to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days

**Table 8-1 (continued)
Sample Preservation and Holding Times^a**

DETERMINATION^a	MATRIX^b	CONTAINER^c	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO ₂ C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S ₂ F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO ₃ B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times^a

DETERMINATION^a	MATRIX^b	CONTAINER^c	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury (EPA 200.7, 200.8, 200.9, 6010, 6020)	W, DW	P,G	HNO ₃ to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury (EPA 245.1, 7470, 7471, 1631E)	W	P,G	HNO ₃ to pH<2	28 days
	S	P,G	Cool, 4°C	28 days
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H ₂ SO ₄ to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO ₃ to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H ₂ SO ₄ to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times^a

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Voiatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace Residual Chlorine Present: 10% Na ₂ S ₂ O ₃ , HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace Residual Chlorine Present: 10% Na ₂ S ₂ O ₃ , HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg Na ₂ S ₂ O ₃ , No Headspace	28 days

**Table 8-1 (continued)
 Sample Preservation and Holding Times^a**

DETERMINATION^a	MATRIX^b	CONTAINER^c	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; ^f 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark ^g	7 days until extraction; ^f 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; ^f 40 days after extraction
Organophosphorus Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C ^g	7 days until extraction; ^f 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; ^f 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H ₂ SO ₄ to pH<2, Cool, 4°C ^g	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH ≥10, Cool, 4°C ^g	30 days until extraction; 30 days after extraction

**Table 8-1 (continued)
 Sample Preservation and Holding Times^a**

DETERMINATION^a	MATRIX^b	CONTAINER^c	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH \leq 2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg Na ₂ S ₂ O ₃ , No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH $<$ 3; 80 mg/L Na ₂ S ₂ O ₃ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/40mL NaS; Cool, $<$ 6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH \leq 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L Na ₂ S ₂ O ₃ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L Na ₂ S ₂ O ₃ , Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH ₄ Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH \leq 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO ₃ to pH $<$ 2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO ₃ to pH $<$ 2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH $<$ 2, No Headspace	14 days until extraction; 14 days after extraction

**Table 8-1 (continued)
 Sample Preservation and Holding Times^a**

DETERMINATION ^a	MATRIX ^b	CONTAINER ^c	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark ^g TCLP extract: Cool, 4°C, Store in Dark ^g	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction

- a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
- b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
- c P = Polyethylene; G = Glass
- d For chlorinated water samples
- e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
- f Fourteen days until extraction for soil, sediment, and sludge samples.
- g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-2

**Columbia Analytical Services, Inc.
 Cooler Receipt and Preservation Form**

PC _____

Client / Project: _____ Service Request **K09**

Received: _____ Opened: _____ By: _____

1. Samples were received via? *US Mail Fed Ex UPS DHL GH GS PDX Courier Hand Delivered*
2. Samples were received in: (circle) *Cooler Box Envelope Other _____ NA*
3. Were custody seals on coolers? *NA Y N* If yes, how many and where? _____
 If present, were custody seals intact? *Y N* If present, were they signed and dated? *Y N*
4. Is shipper's air-bill filed? If not, record air-bill number: _____ *NA Y N*
5. **Temperature of cooler(s) upon receipt (°C):** _____
Temperature Blank (°C): _____
Thermometer ID: _____
6. If applicable, list Chain of Custody Numbers: _____
7. Packing material used. *Inserts Baggies Bubble Wrap Gel Packs Wet Ice Sleeves Other _____*
8. Were custody papers properly filled out (ink, signed, etc.)? *NA Y N*
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* *NA Y N*
10. Were all sample labels complete (i.e analysis, preservation, etc.)? *NA Y N*
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* *NA Y N*
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** *NA Y N*
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* *NA Y N*
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* *NA Y N*
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** *NA Y N*
16. Was C12/Res negative? *NA Y N*

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

*Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).
Additional Notes, Discrepancies, & Resolutions: _____

Figure 8-3 Tier V Form

Client : Project Chemist :
Project Name : Service Request :
Project Number : SMO LimsTemplate ID :
Project Description :

QAPP/SOW Information :

Reporting

Tier Level : PDF: Report to :
in result field use : EDO :
Flagging Requirements :
Other Requirements :

Sample Considerations

Sample Limitations :
Sample Prep/Analysis :
Non-Standard Holdtimes :
Historical Data :
Comments :

9.0 ANALYTICAL PROCEDURES

CAS employs methods and analytical procedures from a variety of sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by CAS is described in SOPs specific to each method. A list of NELAP-accredited methods are given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

CAS strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that CAS has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples, are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

- 1) The number of (field) samples in a batch is not to exceed 20.
- 2) All (field) samples in a batch are of the same matrix.
- 3) The QC samples to be processed with the (field) samples include:
 - a) Method Blank (a.k.a. Laboratory Reagent Blank)
Function: Determination of laboratory contamination.
 - b) Laboratory Control Sample (a.k.a. Laboratory Fortified Blank)
Function: Assessment of method performance
 - c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*
Function: Assessment of matrix bias
 - d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*
Function: Assessment of batch precision

* A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.

- 4) A single lot of reagents is used to process the batch of samples.
- 5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.
- 6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.
- 7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.
- 8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).
- 9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
- 10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
- 11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

CAS not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

CAS commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods(3620, 3630, 3640, 3660, 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at CAS are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Equipment which has been subjected to overloading or mishandling, or has been shown by verification or otherwise to be defective; is taken out of service until it has been repaired. The equipment is placed back in service only after verifying by calibration that the equipment performs satisfactorily. An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC)*. The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by a professional metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on a semi-annual basis.

10.3 Water Purification Systems

CAS uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked at a point downstream of the purification system at a tap in the laboratory, and monitoring documented.

10.4 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A₂LA. CAS relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be "dropped" from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL)*.

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL)*.

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL)*.

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at CAS include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be "dropped" from the resulting calibration curve. Standard CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be "dropped" from the resulting calibration curve. A correlation coefficient of ≥ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis of field samples, acceptable method performance is established by performing demonstration of capability analyses and performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. CAS has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the methodology or are statistically derived based on the laboratory's actual historical data obtained from the various QC measurements for each analytical method. The Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - Where required by mandatory test method, regulation, or accreditation protocols, a demonstration of capability (DOC) is made prior to using any test method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control reference material or quality control sample is obtained. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results or MDL study results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or

caused by an artifact of the measurement system (e.g., contamination). CAS utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources: For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. The current acceptance limits for accuracy and precision are available from the laboratory and on the accompanying CD-ROM. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. CAS has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using CAS or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at CAS/Kelso are determined annually, and may change slightly from year to year. If an MDL study is not performed annually, an MDL verification check is performed quarterly on every instrument used in the analysis. The MDLs are determined by following the *SOP for the Determination of Method Detection Limits and Limits of Detection*, which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using MDL verification samples. The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. the lower limit of quantitation). Therefore, analyses are calibrated to the MRL, or lower. To take into account day-to-day fluctuations in instrument sensitivity, analyst performance, and other factors, the MRL is established at three times the MDL (or greater). The current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. In addition, a number of other quality control processes that may impact analytical results are also described below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < 1/2 MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is(are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$\text{Recovery (\%)} = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.11 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\text{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where S_1 and S_2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.12 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.13 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.14 Control Charting

The generation of control charts is routinely performed at CAS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.15 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

CAS reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process*.

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
 1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
 2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
 1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
 2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.

- GC/MS Methods - Two criteria are used to verify identification:
 1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
 2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, AFCEE QAPP protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications, or a new initial calibration is performed. For DoD projects, the concentration of these two consecutive must be varied as required by the DoD QSM, Version 3.

- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP, may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.
- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP *Confirmation Procedure for GC and HPLC Analysis(SOC-CONF)*. If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – Following sample analysis and data reduction, the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.

- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.
- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. CAS maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The CAS-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the CAS client.

12.5 Documentation

CAS maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, CAS provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, CAS provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. CAS is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of CAS Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of CAS/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of CAS/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices and files, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

CAS/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. CAS routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1 Current CAS Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- Naval Facilities Engineering Service Center Validated Laboratory for NFESC Parameters
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE) Approved Laboratory for AFCEE Projects
- U.S. Army Corps of Engineers Approved Laboratory for USACE Projects
- U.S. EPA Region 8 Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
License No. AZ0339
- State of Arkansas, Department of Environmental Quality
Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
Certification No. 2286
- State of Colorado, Department of Public Health and Environment
Certified Drinking Water Laboratory
- State of Florida, Department of Health
Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
Certified Drinking Water Laboratory
- State of Indiana, Department of Health
Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)
State and Local Programs (continued)

- State of Minnesota, Department of Health
Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at CAS (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at CAS before it may be used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Equipment records also include a copy of the manufacturer's manual(s) and dates and results of calibrations.

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist may be notified in order to ascertain if contact with the client is necessary.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the SOP for Corrective Action (SOP No. ADM-CA). This form is utilized to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Corrective action due to a performance audit or a proficiency(PT) sample finding is initiated by the Quality Assurance Manager; the affected laboratory supervisors and managers are promptly informed of performance audit results requiring corrective action.

Figure 15-1

Columbia Analytical Services, Inc.

Nonconformity and Corrective Action Report

NONCONFORMITY

NCAR No. _____

PROCEDURE (SOP or METHOD): _____	EVENT DATE: _____	
EVENT: <input type="checkbox"/> Missed Holding Time	<input type="checkbox"/> QC Failure	<input type="checkbox"/> Lab Error (spilled sample, spiking error, etc.)
<input type="checkbox"/> Method Blank Contamination	<input type="checkbox"/> Login Error	<input type="checkbox"/> Project Management Error
<input type="checkbox"/> Equipment Failure	<input type="checkbox"/> Unacceptable PT Sample Result	
<input type="checkbox"/> SOP Deviation	<input type="checkbox"/> Other (describe): _____	
SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED		
DETAILED DESCRIPTION		
ORIGINATOR: _____	DATE: _____	
PROJECT MANAGER(S): _____ NOTIFIED BY: _____	DATE: _____	

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity <u>and</u> prevent its recurrence. Include Project Manager instructions here.
Is the data to be flagged in the Analytical Report with an appropriate qualifier? <input type="checkbox"/> No <input type="checkbox"/> Yes

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____	Date: _____
Comments:	
QA PM Verification and Approval of Corrective Action _____	Date: _____
Comments:	
Customer Notified by <input type="checkbox"/> Telephone <input type="checkbox"/> Fax <input type="checkbox"/> E-mail <input type="checkbox"/> Narrative <input type="checkbox"/> Not notified	
Project Manager Verification and Approval of Corrective Action _____	Date: _____
Comments:	
(Attach record or cite reference where record is located.)	

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a case narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and corrected if possible.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

Any operational or quality assurance problems noted by the Laboratory Director are then addressed during the senior staff operations meetings with all appropriate department managers. The Laboratory Director also performs a documented management review annually of the quality and management systems to identify any necessary changes or improvements to the quality system or quality assurance policies.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at CAS are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at CAS when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at CAS. Safety training begins with the reading of the *Environmental Health and Safety Manual*. Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer. Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s).

Each employee participates in Ethics training, which is part of the CAS Improper Practices Prevention Program. CAS also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of proficiency. Where the analyst performs the entire procedure, a generic training plan may be used. In cases where work cells are used, a training plan specific to the work cell is established.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
 - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
 - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
 - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

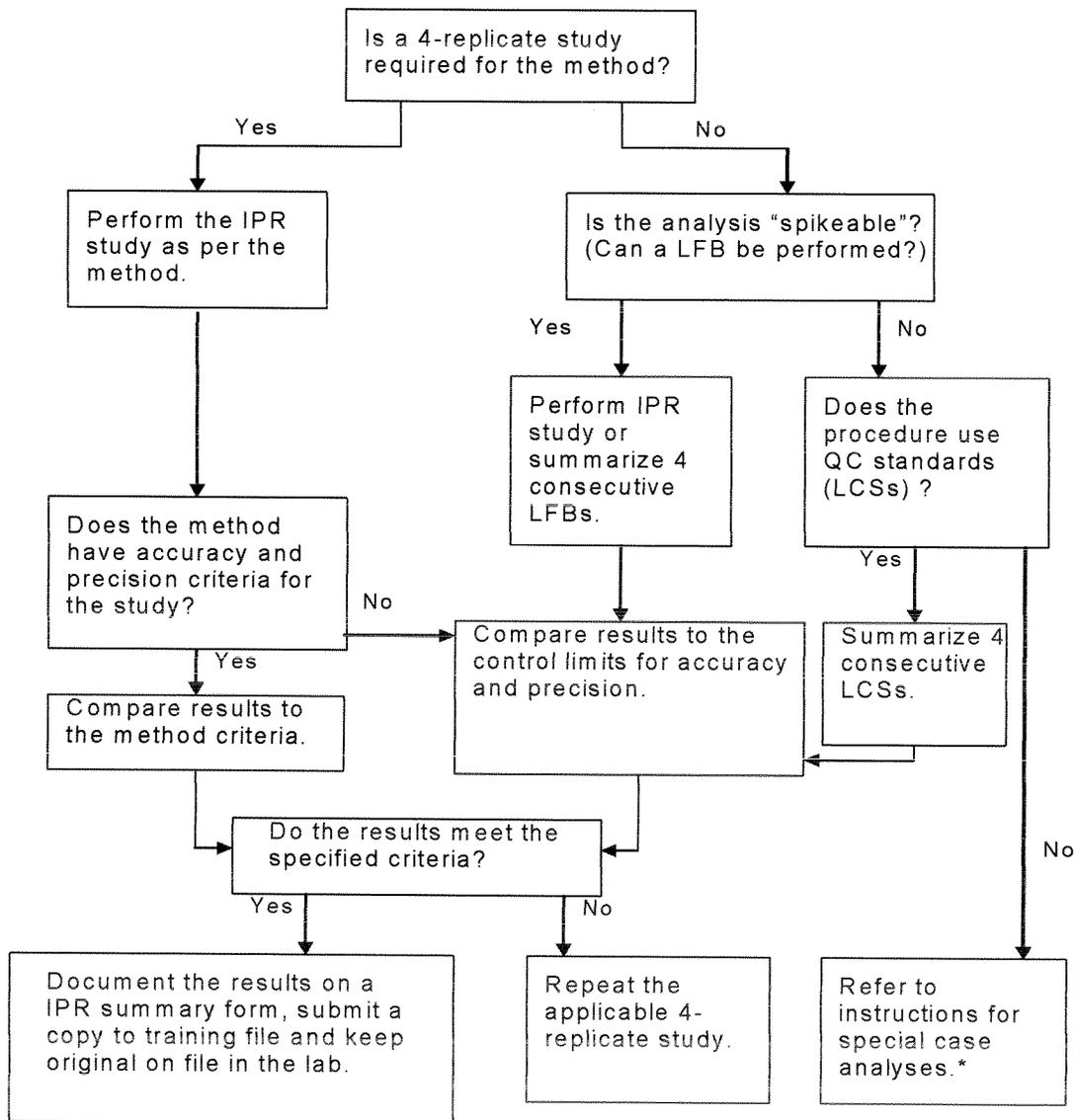
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind PT sample analyses using the test method, or a similar test method using the same technology.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and CAS resumes. A database is used to record the various technical skills and training acquired while employed by CAS. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC)*.

**Figure 17-1
 Initial Demonstration of Capability Requirements^a**



^a For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements

*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.

*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.

* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at CAS are taken from the following references:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.
- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).

- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste*, California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

and

STANDARD OPERATING PROCEDURES

Quality Assurance Manual	2/6/2009
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
Standard Operating Procedure Database	TrainDat.mdb
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CONTROL LIMITS	ADM-CTRL_LIM
CORRECTIVE ACTION	ADM-CA
DATA RECALL	ADM-DATARECALL
HANDLING CUSTOMER FEEDBACK	ADM-FDBK
DETERMINATION OF METHOD DETECTION LIMITS AND LODS	ADM-MDL
DOCUMENT CONTROL	ADM-DOCCTRL
DOCUMENTATION OF TRAINING	ADM-TRANDOC
ELECTRONIC DATA AUDITING	ADM-E_DATAAUDIT
ESTIMATION OF UNCERTAINTY OF MEASUREMENTS	ADM-UNCERT
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY
MANAGERIAL REVIEW OF THE LABORATORY'S QUALITY SYSTEM	ADM-MGMTRVW
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT
PREPARATION OF ELECTRONIC DATA FOR ORGANIC ANALYSES ELECTRONIC DATA AUDITS	ADM-EDATA
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP
PROFICIENCY TESTING SAMPLE ANALYSIS	ADM-PTS
PURCHASING THROUGH CAS PURCHASING DEPARTMENT IN KELSO	ADM-PUR
QUALIFICATION OF SUBCONTRACT LABORATORIES OUTSIDE OF CAS NETWORK	ADM-SUBLAB

SIGNIFICANT FIGURES	ADM-SIGFIG
CONFIRMATION of ORGANIC ANALYTE IDENTIFICATION and QUANTITATION	ADM-CONFIRM
PREVENTATIVE ACTION	ADM-PA

<u>ADMINISTRATIVE – LOCAL LABORATORY</u>	<u>FILE NAME</u>
CHECKING PIPETTE CALIBRATION	ADM-CPIP
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT
DATA ARCHIVING	ADM-ARCH
DATA REPORTING AND REPORT GENERATION	ADM-RG
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP
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LABORATORY BALANCE MONITORING AND CALIBRATION	ADM-BAL
LABORATORY DATA REVIEW PROCESS	ADM-DREV
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC
PROJECT MANAGEMENT	ADM-PCM
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
SAMPLE BATCHES	ADM-BATCH
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SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
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<u>TECHNICAL STANDARD OPERATING PROCEDURES</u>	
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COLUMBIA ANALYTICAL SERVICES, INC. , KELSO, WA.
STANDARD OPERATING PROCEDURES TABLE OF CONTENTS

January 19, 2009

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COLIFORM, FECAL	BIO-9221FC
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COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT® and COLITAG	BIO-9223
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COLILERT® COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT
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SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
EPA CLP ORGANICS ANALYSES	CLP_ORGA
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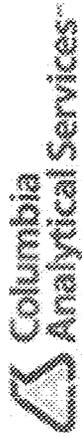
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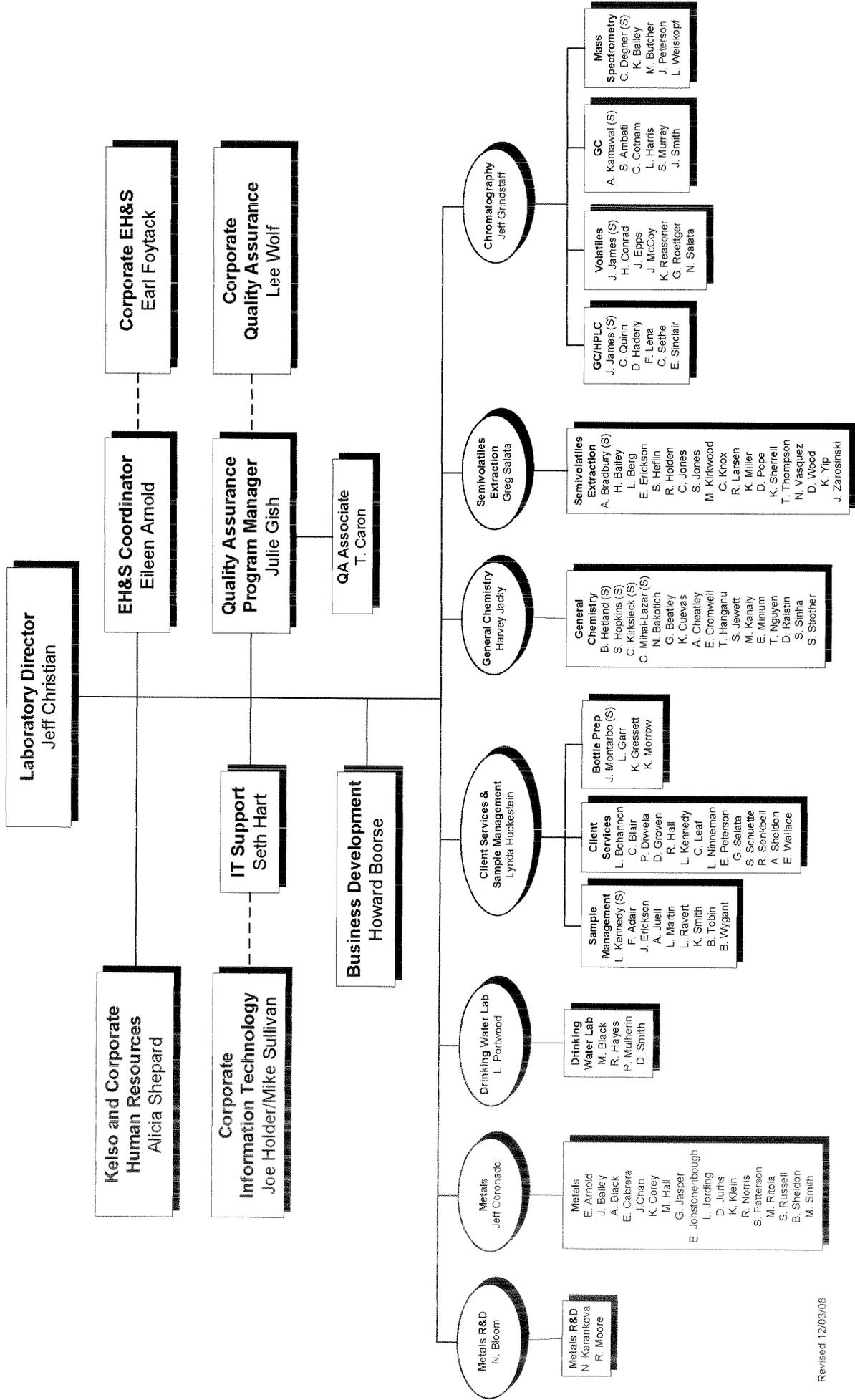
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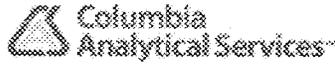
APPENDIX B

**ORGANIZATIONAL CHARTS and RESUMES OF KEY
PERSONNEL**

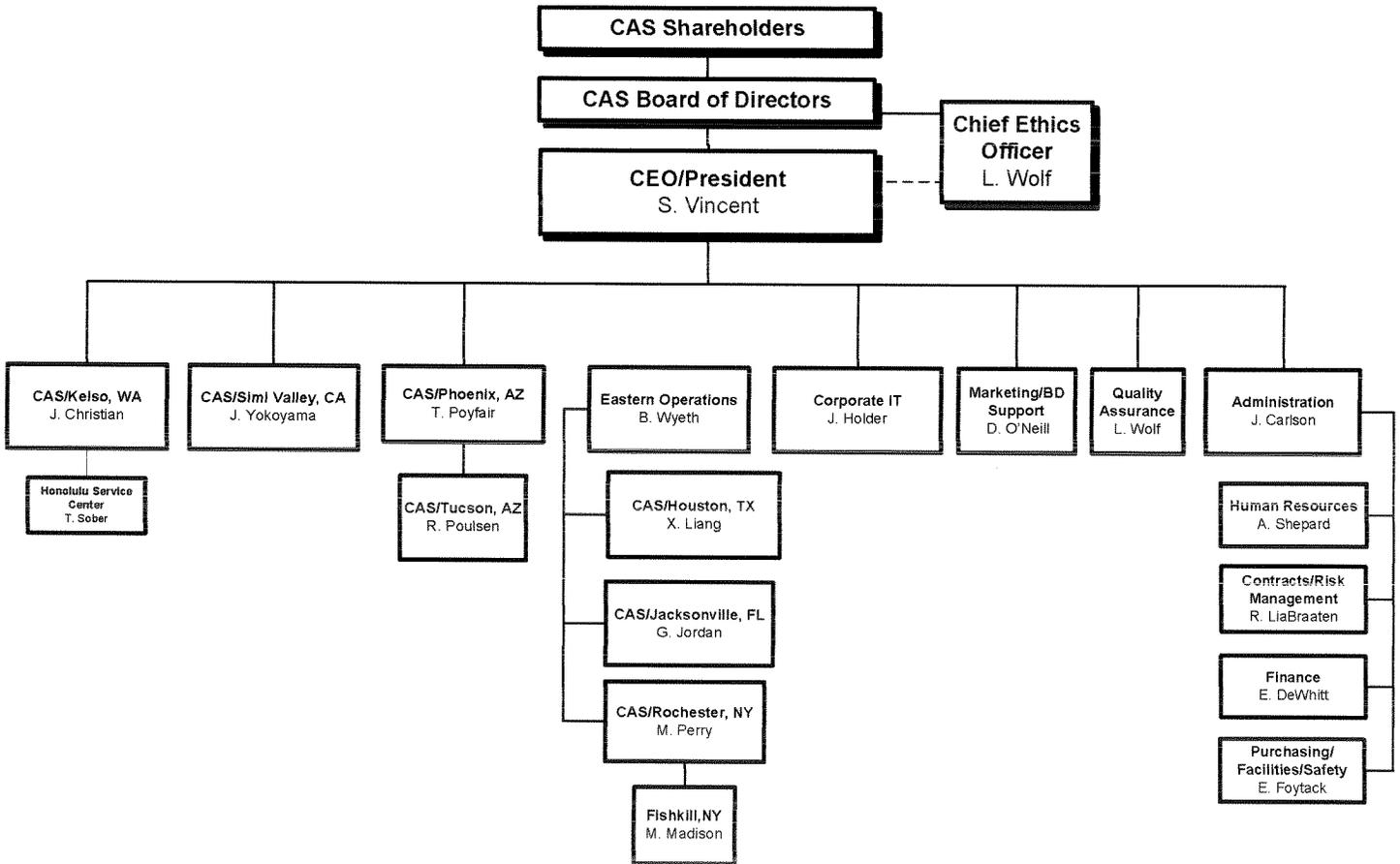


Environmental and General Testing Division Kelso, Washington Laboratory Organization





Laboratory Division Organization



JEFFREY D. CHRISTIAN
1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT/NW REGIONAL DIRECTOR – 1996 to Present

Responsibilities

Responsible for all phases of laboratory operations at the Kelso (WA) and Redding (CA) facilities, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.

Experience

Laboratory Director, Kelso Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Operations Manager, Kelso Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts.

Project Chemist and Manager, Metals Analysis Laboratory, Columbia Analytical Services, Kelso, Washington, 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories.

Scientist, Weyerhaeuser Technology Center, Federal Way, Washington, 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods.

Lead Technician, Metals Lab, Weyerhaeuser Technology Center, Federal Way, Washington, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing.

Research Assistant, ITT Rayonier, Olympic Research Division, Shelton, Washington, 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.

Education

B.S., Chemistry, Evergreen State College, Olympia, Washington, 1993.

ICP/MS Training Course, VG-Elemental, 1992.

Coursework, Pacific Lutheran University, Tacoma, Washington, 1988-1989.

Coursework, Tacoma Community College, Tacoma, Washington, 1970-1971, 1988-1989.

Perkin-Elmer Advanced Furnace, Norwalk, Connecticut, 1986.

CERTIFICATION, Chemistry, L.H. Bates Technical, Tacoma, Washington, 1978.

Coursework, Central Washington University, Ellensburg, Washington, 1969-1970.

**Publications/
Presentations**

On request.

JULIE GISH
1996 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER – 2008 to Present
Responsibilities	Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits and provides audit responses and initiates any corrective actions. Coordinate the analysis and reporting of PT samples. Conduct internal audits and make recommendations for corrective action.
Experience	<p>Scientist IV, Semi-Volatile Mass Spectrometry Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.</p> <p>Technical Manager I, Semi-Volatile GC Organics Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.</p> <p>Scientist III, Semi-Volatile Organics Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.</p> <p>Scientist, Volatile Organics Sample Preparation, Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility, 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).</p> <p>Organics Chemist and GC/MS Chemist, Coffey Laboratories, Portland, Oregon, 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.</p> <p>QC Manager/QC Supervisor and Product Manager, Corn Products, Frito-Lay, Inc., Vancouver, Washington, 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.</p> <p>Food Technologist, QA Department, Kraft, Inc., Buena Park, California, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.</p>
Education	MS, Food Science, Minor in Industrial Engineering, Oregon State Univ. Corvallis, Oregon, 1978. BS, Food Science, Minor in Business Administration, Utah State University, Logan, Utah, 1975
Publications/ Presentations	<p><i>Quality Improvement Team Leader</i>, Coffey Laboratories, Portland, Oregon. 1991</p> <p><i>Methods Improvement Program</i>, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.</p> <p><i>Statistical Process Control and Total Quality Management</i>, Frito-Lay, Vancouver, Washington. <i>Routine Training Classes 1986-1988</i></p>

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

CLIENT SERVICES MANAGER IV – 1998 to Present

Responsibilities

Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance as well as project organization to work received by the laboratory.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, Columbia Analytical Service, Inc., Kelso, Washington, 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as well as project organization to work received by the laboratory

Project Chemist and Department Manager, General Chemistry Laboratory, Columbia Analytical Services, Inc., 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.

Analyst III, Columbia Analytical Services, Inc., Kelso, Washington, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.

Microbiologist/Chemist, Coffey Laboratories, Portland, Oregon, 1983. Coliform analysis; water chemistry.

Laboratory Assistant, Oregon State University, Corvallis, Oregon, 1983. Wheat spike dissection and tissue culture.

Education

BS, Microbiology, Oregon State University, Corvallis, Oregon, 1983.

JEFFREY A. CORONADO
1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, METALS DEPARTMENT MANAGER – 2001 to Present

Responsibilities

Primary responsibilities include management of the Metals laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the Metals department. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Metals Department Manager, Columbia Analytical Services, Inc., Kelso, Washington, 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.

Supervisor, GFAA Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.

Education

Field Immunoassay Training Course, EnSys Inc., 1995.

Winter Conference on Plasma Spectrochemistry, San Diego, California, 1994.

ICP-MS Training Course, VG-Elemental, 1992.

BS, Chemistry, Western Washington University, Bellingham, Washington, 1988.

BA, Business Administration, Western Washington University, Bellingham, Washington, 1985.

JEFFREY A. GRINDSTAFF
1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, CHROMATOGRAPHY AND MASS SPECTROMETRY LABORATORIES – 1997 to Present

Responsibilities

Primary responsibilities include management of the GC/MS SemiVoa and VOA laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the GC/MS departments. Also responsible for departmental budgets, method development efforts, and resource allocation. Also performs GC/MS maintenance and troubleshooting.

Documentation of Demonstration of Capabilities is available for review.

Experience

Manager, GC/MS VOA Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.

Scientist III, GC/MS VOA Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.

Chemist, Enseco-CRL, Ventura, California, 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.

GC/MS Chemist, VOA Laboratory Coast-to-Coast Analytical Service, San Luis Obispo, California, 1990-1991. Responsible for standard preparation for VOA analyses and instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.

Education

Mass Selective Detector Maintenance, Hewlett-Packard Education Center, 1993.

Interpretation of Mass Spectra I, Hewlett-Packard Analytical Education Center, 1992.

B.S., Chemistry, California Polytechnic State University, San Luis Obispo, California, 1989.

A.A., Liberal Arts, Allan Hancock College, Santa Maria, California. 1986

**Publications/
Presentations**

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, Florida. 1996

Affiliations

American Chemical Society. 1989

HARVEY L. JACKY
1999 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	TECHNICAL MANAGER II – 2008 to Present
Responsibilities	Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. Documentation of Demonstration of Capabilities is available for review.
Experience	<p>Project Manager III, Columbia Analytical Services, Inc., Kelso, WA, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.</p> <p>Director of Project Management, Coffey Laboratories, Portland, Oregon, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy.</p> <p>Project Manager/Chemist, Coffey Laboratories, Portland, Oregon, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon.</p> <p>Technical Sales Representative, Coffey Laboratories, Portland, Oregon, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management.</p> <p>Senior Chemist/Laboratory Chemical Hygiene Officer, Coffey Laboratories, Portland, Oregon, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.</p>
Education	<p>40-Hour Hazmat Certification, PBS Environmental, 1996.</p> <p>Industrial Emergency Response, SFSP Seminar, 1991</p> <p>BS, Zoology, Oregon State University, Corvallis, Oregon, 1988.</p> <p>BS, General Science, Oregon State University, Corvallis, Oregon, 1988.</p> <p>COURSEWORK, General Studies, Linfield College, McMinnville, Oregon, 1981-1982.</p>
Publications/ Presentations	<p><i>Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System.</i> Biogeochemistry, Portland State University, 1996</p>

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position	SCIENTIST IV, DRINKING WATER LABORATORY MANAGER – 2008 to Present
Responsibilities	Responsible for the overall operation and supervision of the Organic Drinking Water department, including oversight of UCMR2 analyses. Perform analyses and conduct data review. Perform method development. Work with project management of drinking water accounts. Development of Standard Operating Procedures for drinking water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC. Documentation of Demonstration of Capabilities is available for review.
Experience	Scientist IV, Drinking Water Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, QC, and data reporting. Also responsible for preparation of SOPs; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, Primary responsibilities included oversight of the PHC laboratory, including initiating new processes and staff development and training. Responsible for CAS QA compliance, routine system checks. Technical mentor to PHC staff. Also duties listed below under Scientist II and Scientist III. Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below. Scientist II, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Duties primarily as listed below, and including HPLC methods 8310, 8315, and 8330. Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses. Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included performing a wide range of organics extractions and cleanups for water, soil, and oil to be analyzed in the GC, GC/MS, and PHC laboratories. Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. Developed testing which was accredited by the EPA, including metal digestions, phosphates, and TSS/ TDS.
Education	BS, Chemistry, Emphasis in Biochemistry, Whitworth College, Spokane, Washington, 1990. Several vendor chromatography, GC, HPLC, and Quality training courses, 1993-2002.

EILEEN M. ARNOLD
1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFETY OFFICER – 1994 to Present

Responsibilities

Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Chemist, Client Services Group, Kelso Health and Safety Officer, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Scientist IV, Metals Laboratory, Health and Safety Officer, Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992. Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.

Chemist, Dow Corning Corporation, Springfield, Oregon, 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon.

Chemist, Ametek, Inc., Harleysville, Pennsylvania, 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques.

Chemist, Janbridge, Inc., Philadelphia, Pennsylvania, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.

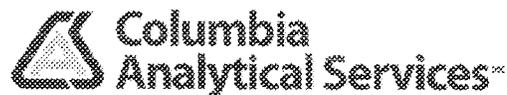
Education

BA, Chemistry, Immaculata College, Immaculata, Pennsylvania, 1977.

Affiliations

American Chemical Society, Member since 1987.

GREGORY G. SALATA
2003 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	PROJECT/EXTRACTIONS MANAGER V – 2003 to Present
Responsibilities	Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows. 2003-Present.
Experience	<p>Project Manager, B&B Laboratories, College Station, Texas, 1999-2003. Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.</p> <p>Graduate Student, Texas A&M University, College Station, Texas, 1991-1999. While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.</p> <p>Analytical Chemist, Science Applications International (SAIC), San Diego, California, 1989-1990. Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.</p> <p>GC Chemist, Analytical Technologies, San Diego, California, 1987-1989. Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.</p>
Education	<p>Ph.D., Oceanography, Texas A&M University, College Station, Texas. 1999 MS, Oceanography, Texas A&M University, College Station, Texas. 1993 BA, Chemistry, University of California San Diego, Revelle College, La Jolla, California. 1987</p>
Publications/ Presentations	<i>Dr. Salata has a number of publications and published abstracts. For a list of these publications and published abstracts, please contact CAS.</i>
Affiliations	American Chemical Society

LEE E. WOLF
1988 TO PRESENT



Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	QUALITY ASSURANCE DIRECTOR AND CHIEF QUALITY OFFICER – 2008 to Present
Responsibilities	Directing the overall corporate-wide quality systems and ethics programs for all CAS facilities. Responsible for ensuring that CAS quality systems and data integrity standards are implemented at all facilities. Act as liaison with government entities involving quality, technical and operational issues. Provide QA input and policy as needed for operations, development initiatives, special projects, planning, and information technology implementation. Provide assistance to QA Program Managers.
Experience	<p>Technical Manager IV, Quality Assurance Program Manager, Columbia Analytical Services, Inc., Kelso, Washington – 2002 to 2008. As part of the management team, responsibilities included the overall management and implementation of the laboratory QA program. This included maintaining accreditations and certifications, and maintaining all necessary documents (QA Manual, SOPs, and QA records). Acted as primary point of contact during laboratory audits and provided audit responses and corrective actions. Coordinated performance audits (PE/PT testing) and conducted internal audits.</p> <p>Scientist IV, Quality Assurance Program Manager, Columbia Analytical Services, Inc., Kelso, Washington, 1996-2002. Duties primarily as listed above.</p> <p>Project Chemist/Principal Organic Scientist, Columbia Analytical Services, Inc., Kelso, Washington, 1994-1996. Responsibilities included GC and GC/MS method development and special projects coordination. Acts as technical advisor to the GC and GC/MS laboratories and GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions, including management of projects for clients, identifying client needs, and preparation of data reports.</p> <p>Semivolatile Organics Department Manager, Columbia Analytical Services, 1988-1994. Responsibilities included overall management of the department. Supervised GC/MS analyses, data review, reporting and related QA/QC functions. Responsible for supervision of staff, training, and scheduling. Beginning in 1992, responsibilities included being a Project Chemist for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client requirements, and preparing data reports.</p> <p>GC/MS Chemist, U.S. Testing Co., Richland, Washington, 1985-1988. Responsibilities included GC and GC/MS analysis of water and soil samples for volatiles and semivolatiles by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Also performed HPLC analysis and pesticide analysis by GC using EPA Methods.</p> <p>Laboratory Assistant, Eastern Washington University, Cheney, Washington, 1985. Responsibilities included supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety.</p>
Education	<p>Pharmaceutical Laboratory Control Systems, Univ. of Wisconsin Short Course, Las Vegas, 2004</p> <p>Test Method Validation in Pharmaceutical Development and Production, Univ. of Wisconsin Short Course, Las Vegas, 2004</p> <p>Documenting Your Quality System, A2LA Short Course, Las Vegas, Nevada, 1998.</p> <p>Internal Laboratory Audits, A2LA Short Course, Las Vegas, Nevada, 1998.</p> <p>Mass Spectra Interpretation, ACS Short Course, Denver, Colorado, 1992.</p> <p>BS, Chemistry, Eastern Washington University, Cheney, Washington, 1985.</p>
Publications/ Presentations	<p><i>Selected Ion Monitoring: Issues for Method Development</i>, Panel Discussion, Association of Official Analytical Chemists, (AOAC) Pacific Northwest Regional Meeting, 1995.</p> <p><i>Method Enhancement Techniques for Achieving Low level Detection of Butyl Tin in Marine Sediments and Tissues</i>, Association of Official Analytical Chemists (AOAC) Pacific Northwest Regional Meeting, 1994.</p> <p><i>The Determination of Low-Level Concentrations of Polynuclear Aromatic Hydrocarbons (PAHs) in Soil and Water Using Gas Chromatography/Mass Spectroscopy Selected Ion Monitoring (GC/MS SIM)</i>, HazMat West, Long Beach, California, 1992.</p>

STEPHEN W. VINCENT

1986 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

PRESIDENT, CAS HOLDINGS INC. – 1986 to Present

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager, Weyerhaeuser Company, Federal Way, Washington, 1979-1986. Responsibilities involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately \$2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist, Weyerhaeuser Company, Longview, Washington, 1975-1979.

Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Market Strategy for Technology Based Companies, Executives Program, Stanford University, 1994.

Advanced Technical Management Program, University of California at Los Angeles, Department of Business, Engineering and Management, 1991.

Completion of Coursework for MS, Pulp and Paper Technology, University of Washington, Seattle, Washington, 1984.

Post Graduate Coursework, Engineering and Management, University of California at Los Angeles, Graduate School of Engineering and Applied Science, Los Angeles, California, 1981.

BS, Oceanography, University of Washington, Seattle, Washington, 1974.

Publications/ Presentations

Mr. Vincent has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Affiliations

American Chemical Society.

Technical Association of the Pulp and Paper Industry.

APPENDIX C
MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balances (10): Precisa and Mettler models	1988-2008	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2): Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2): YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5): COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11): Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2): ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2): Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4) Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5) Fisher Scientific Accumet Model 50	1997	LM	6
Fisher Scientific Accumet Model 25	1993	LM	6
Fisher Scientific Accumet Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2): Fisher Scientific Accumet Model 20	1993	LM	6
Fisher Scientific Accumet Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Perkin Elmer Lambda 25	2008	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance (4) Mettler PM480, AE166, BB300 Ohaus EP613	1999 - 2005 2006	MM MM	18 18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2) Fisher Model 655G VWR Model 1305U	1991 1999	LM LM	18 18
Evaporators (14): Organomation N-Evap (7) Organomation S-Evap (7)	1989-98, 2001, 2006 1989-1991, 2006	LM LM	18 18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52): Branson Model 450 Sonifier (2) Tekmar Sonicator Fisher Scientific Sonicator Soxhtherm (48)	1991 1994 1994 2000, 2008	LM LM LM LM	6 6 6 8
Extractors, TCLP (10): Millipore TCLP Zero Headspace Extractors (10) TCLP Extractor - Tumbler (12 position)	1987-1992 1989	LM LM	2 2
Gel Permeation Chromatography (GPC) (5) ABC single column (3) ABC Autoprep 1000 J2 Scientific	1998, 1999, 2007 1995 2005	LM LM LM	4 4 4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (8) – Horizon SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler AT 250	1989	MM	7
Chromatography Data Systems (12)			7
HP Enviroquant (8)	1994-2002	LM	
Thruput Target (4)	1998-2000	LM	
Gas Chromatographs (11):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (4)	1990 – 1995	LM	7
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	7
Agilent 7890A Dual ECD Detectors	2008	LM	7
Agilent 7683B autosampler			

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	5
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS –			
Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (6):			
Hewlett-Packard 5890 Series II with PID/PID/FID(2)	1991	LM	4
EST-ENCON Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass(2) Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (10) – Horizon SPE-DEX Solid Phase Extractor	2003/2008	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization:	2003	LM	2
UV/Fluorescence detectors	2003	LM	2
Pickering PCX-5200 Post-column derivitization unit	2003	LM	2
Agilent 6890N GC/Dual ECD system w/ autosamplers	2003	LM	2
Agilent 7890 GC/Dual ECD w/autosamplers	2008	LM	2
Varian Ion trap GC/MS:	2003	LM	2
Varian 3800 GC w/CP8400 autosampler	2006	LM	2
Varian Saturn 2100T mass spectrometer	2003	LM	2
Thremo Ion Trap GC/MS w/TriPlus autosampler	2008	LM	2

Metals Method Development Laboratory			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Perkin-Elmer ICP/MS Elan 9000 w/ Perkin-Elmer AS-93+ Autosampler	2008	LM	2
Perkin-Elmer Series 200 IC	2008	LM	2
Brooks Rand III Atomic Fluorescence Spectrophotometer - 2	2008	LM	2
Oriel Atomic Fluorescence Spectrophotometer – Lab Designed	2008	LM	2
Balances - 4	2008	LM	2
Ovens - 2	2008	LM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Forma Scientific Bio Freezer	2008	LM	2
Digital Shaker SK-71	2008	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 180 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro- photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro- photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency
ICP - MS	Check argon dewar Check water level in chiller Complete instrument log Replace peristaltic pump tubing Clean sample and skimmer cones Clean RF contact strip Inspect nebulizer, spray chamber, and torch Clean lens stack/extraction lens Check rotary pump oil Change rotary pump oil	Daily Daily Daily Daily As needed As needed Clean as needed As needed Monthly Every six months
Gel-Permeation Chromatographs	Clean and repack column Backflush valves	As needed As needed
High Pressure Liquid Chromatographs	Backflush guard column Backflush column Change guard column Change column Change in-line filters Leak check Change pump seals Change pump diaphragm Clean flow cell Fluorescence detector check Diode array absorbance check	As needed As needed As needed when back pressure too high Annually or as needed As needed After column maintenance As needed Annually As needed Daily Daily
Gas Chromatographs, Semivolatiles	Check gas supplies Change in-line filters Change septum Change injection port liner Clip first 6-12" of capillary column Change guard column Replace analytical column Check system for gas leaks Clean FID Clean ECD Leak test ECD	Daily, replace if pressure reaches 50psi Quarterly or after 30 tanks of gas Daily Weekly or as needed As needed As needed As needed when peak resolution fails After changing columns and after any power failure Weekly or as needed Quarterly or as needed Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies Change in-line filters Change septum Change injection port liner Clip first 6-12" of capillary column Change guard column Replace analytical column Clean source Change pump oil	Daily, replace if pressure reaches 50psi Annually or as needed Daily, when in use Weekly or as needed As needed As needed As needed when peak resolution fails As needed when tuning problems As specified by service specifications
Purge and Trap Concentrators	Change trap Change transfer lines Clean purge vessel	Every four months or as needed Every six months or as needed Daily
Gas Chromatographs, Volatiles	Check gas supplies Change in-line filters Change septum Clip first 6-12" of capillary column Change guard column Replace analytical column Check system for gas leaks Clean PID lamp Clean FID Change ion exchange resin Replace nickel tubing	Daily, replace when pressure reaches 50 psi Quarterly or after 30 tanks of gas Daily As needed As needed As needed when peak resolution fails After changing columns and after any power failure As needed As needed Every 60 days Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies Change in-line filters Change septum Clip first foot of capillary column Change guard column Replace analytical column Clean jet separator Clean source Change pump oil	Daily, replace when pressure reaches 50 psi Annually or as needed Daily As needed As needed As needed when peak resolution fails As needed As needed when tuning problems As specified by service specifications

APPENDIX E
LIST OF NELAC ACCREDITED METHODS

APPENDIX F

ADDITIONAL AGENCY-SPECIFIC DOCUMENTS